

REPORT DOCUMENTATION PAGE

Form Approved
OMB NO. 0704-0188

Public reporting burden for this collection of information is estimated to average 1 hour per response, including the time for reviewing instructions, searching existing data sources, gathering and maintaining the data needed, and completing and reviewing the collection of information. Send comment regarding this burden estimate or any other aspect of this collection of information, including suggestions for reducing this burden, to Washington Headquarters Services, Directorate for Information Operations and Reports, 1215 Jefferson Davis Highway, Suite 1204, Arlington, VA 22202-4302, and to the Office of Management and Budget, Paperwork Reduction Project (0704-0188), Washington, DC 20503.

1. AGENCY USE ONLY (Leave blank)	2. REPORT DATE	3. REPORT TYPE AND DATES COVERED Final Progress	
10 Oct. 96		5. FUNDING NUMBERS DAAH04-94-G-0070	
4. TITLE AND SUBTITLE Superplasticity at High Strain Rates in Aluminum Alloys		6. AUTHOR(S) Kenji HIGASHI	
7. PERFORMING ORGANIZATION NAMES(S) AND ADDRESS(ES) Osaka Prefecture University 1-1, Gakuen-cho, Sakai, Osaka 593 Japan		8. PERFORMING ORGANIZATION REPORT NUMBER	
9. SPONSORING / MONITORING AGENCY NAME(S) AND ADDRESS(ES) U.S. Army Research Office P.O. Box 12211 Research Triangle Park, NC 27709-2211		10. SPONSORING / MONITORING AGENCY REPORT NUMBER ARO 33027.4-MS	
11. SUPPLEMENTARY NOTES The views, opinions and/or findings contained in this report are those of the author(s) and should not be construed as an official Department of the Army position, policy or decision, unless so designated by other documentation.			
12a. DISTRIBUTION / AVAILABILITY STATEMENT Approved for public release; distribution unlimited.		12 b. DISTRIBUTION CODE	
13. ABSTRACT (Maximum 200 words) The detailed experimental data of microstructural developments during the optimum thermomechanical treatments and superplastic properties in the wide deformation range have been investigated on fourteen P/M aluminum alloys with various chemical matrix compositions and different typed precipitate particles. It was revealed that the differences in both size and volume among the second phase particles, which increase with content of Zr, Cr, Mn or Y, affect on the recrystallization behavior, grain size and superplastic properties of these aluminum alloys. Some of them exhibited high-strain-rate superplasticity, so the optimizing processing methods in this proposal were very powerful to provide the desired structures required for high-strain-rate superplasticity in these aluminum alloys. A theoretical interpretation, based on the experimental data by tensile test and the observed grain sizes, has been revealed that values in activation energy for all alloys are between 140 and 155 kJ/mol ⁻¹ , which are similar to that for lattice self-diffusion of aluminum. Each mechanical data of each alloy can be presented by a single equation. It was postulated that superplastic flow in these P/M aluminum alloys was fundamentally controlled by a grain boundary sliding mechanism accommodated by dislocation climb controlled by lattice self-diffusion. However, for the statically recrystallized alloys consisted of the high-angle grain boundaries, the Dorn typed equation presents n=2, p=2 and D=D _L , whereas for the dynamically recrystallized alloys with the low-angle grain boundaries, n=3, p=2 and D=D _L . The consideration for deformation mechanisms with the accommodation helper by such a liquid has been applied on the aluminum alloys, but it remained unclear. The contribution by an accommodation helper to high-strain-rate superplasticity will be the further research subjects, including the investigation of cavitation and fracture.			
14. SUBJECT TERMS 1 May. 94 ~ 30 September. 96			15. NUMBER OF PAGES 15
			16. PRICE CODE
17. SECURITY CLASSIFICATION OR REPORT UNCLASSIFIED	18. SECURITY CLASSIFICATION OF THIS PAGE UNCLASSIFIED	19. SECURITY CLASSIFICATION OF ABSTRACT UNCLASSIFIED	20. LIMITATION OF ABSTRACT UL

NSN 7540-01-280-5500

Standard Form 298 (Rev. 2-89)
Prescribed by ANSI Std. Z39-18
298-102

**SUPERPLASTICITY AT HIGH STRAIN RATES
IN ALUMINUM ALLOYS**

FINAL PROGRESS REPORT

KENJI HIGASHI

10 OCTOBER 1996

P-33027-MS

**DAAH04-94-G-0070
P00001 & P00002**

19970210 116

OSAKA PREFECTURE UNIVERSITY

**APPROVED FOR PUBLICATIONS;
DISTRIBUTION UNLIMITED**

**THE VIEWS, OPINIONS AND/OR FINDINGS
CONTAINED IN THIS REPORT ARE THOSE OF THE
AUTHOR AND SHOULD NOT BE CONSTRUED AS
AN OFFICIAL DEPARTMENT OF THE ARMY
POSITION, POLICY OR DECISION UNLESS SO
DESIGNATED BY OTHER DOCUMENTATION."**

1. FOREWORD

This research program (Grant No. DAAH04-94-G-0070, P00001&00002) designed to investigate superplasticity at high strain rates in aluminum alloys. This program was initiated in order to investigate the understanding of the microstructural development to fine grained superplastic structures by thermomechanical processing and the role of microstructural evolution on the subsequent mechanical properties in fourteen P/M Al-Mg and Al-Cu system alloys including Cr, Mn, Y and Zr.

The investigating experiments consisted of three parts;

1. studying the determination of main alloy compositions of P/M aluminum alloys can exhibit the superplasticity at high strain rates,
2. studying the possibility of grain refinement by an optimum rout of thermo-mechanical treatment in each P/M aluminum alloy, including the observation for the microstructural developments during the optimum thermomechanical treatment,
3. studying the microstructural evolution during superplastic flow at high strain rates and its relating changes of the mechanical properties, including a determination of the flow stress and the strain rate sensitivity exponent.

Based on the detailed experimental measurements of the mechanical properties by tensile test over a wide deformation range and the microstructural information, the clarification of mechanisms of high-strain-rate superplasticity in P/M aluminum alloys has been tried. Also a theoretical interpretation of the data has been expanded by the new concept of the developing model for deformation mechanisms with the accommodation helper by a liquid phase.

The final objective performed in the present final progress report is to summarize many investigated experiments, to provide a good understanding of the changes in mechanical properties, and to develop the deformation mechanism for high-strain-rate superplasticity.

2. TABLE OF CONTENTS

1. FOREWORD

2. TABLE OF CONTENTS

3. LIST OF ILLUSTRATIONS AND TABLES

4. RESULTS

A. STATEMENT OF THE RESEARCH

- (1) Determination of Chemical Compositions of P/M Alloys
- (2) Observation of Recrystallization Behavior for As-extruded Materials
- (3) Microstructural Evolution and Quantitative Metallographic Measurements
- (4) Experimental Measurements of Superplastic Properties and Grain Sizes
- (5) Theoretical Interpretation to Superplastic Deformation Mechanisms

B. SUMMARY

C. LIST OF THE PUBLICATIONS

D. LIST OF PARTICIPATING SCIENTIFIC PERSONNEL

5. REPORT OF INVENTIONS: **NONE**

6. BIBLIOGRAPHY: **NONE**

3. LIST OF ILLUSTRATIONS AND TABLES

Table 1: The final chemical compositions of P/M aluminum alloys (wt%).

Table 2: Superplastic properties of P/M aluminum alloys.

Table 3: A summary of the optimum superplastic temperatures, the solidus temperatures and the incipient melting temperatures of a number of P/M aluminum alloys.

Fig. 1: The changes of the grain sizes in P/M aluminum alloys with contents of additional elements of Cr, Mn, Y and Zr.

Fig. 2: Grain size dependence of superplastic strain rate in P/M aluminum alloys.

Fig. 3: Diffusion-compensated strain rate versus modulus-compensated stress for all P/M aluminum alloys.

4. RESULTS

A. STATEMENT OF THE RESEARCH

The outlines of the investigations in the present research are the following;

(1) Determination of Chemical Compositions of P/M Alloys

(TPR No.1; 1 May 94 ~ 10 July 94)

The chemical compositions of P/M aluminum alloys were nominated in the present work are given as Al-5wt%Cu-Xwt%Zr [0.1<X<1], Al-5wt%Mg-Ywt%Cr [1<Y<2], Al-5wt%Mg-Zwt%Mn [1<Z<3], Al-5wt%Mg-Wwt%Y [0.1<W<2] and Al-5wt%Mg-Vwt%Zr [0.1<V<1]. The characteristics of the precipitates were changed easily by changing the contents of Zr, Cr, Y and Mn. Finally the chemical compositions of two systems P/M aluminum alloys (Al-4.5wt% Cu and Al-5wt%Mg alloys), as shown in **Table 1**, were selected for using in the present investigation.

Table 1: The final chemical compositions of P/M aluminum alloys (wt%).

Alloy	Cu	Mg	Zr	Cr	Mn	Y	Fe	Si
Al-4.5Cu-0.2Zr	4.33		0.18				0.02	0.030
Al-4.5Cu-0.3Zr	4.29		0.32				0.03	0.030
Al-4.5Cu-0.8Zr	4.29		0.81				0.07	0.070
Al-5Mg-0.2Zr		4.80	0.22			<0.01	0.007	
Al-5Mg-0.5Zr		4.82	0.48			<0.01	0.007	
Al-5Mg-0.8Zr		4.76	0.84			<0.01	0.011	
Al-5Mg-0.4Cr		4.78		0.36		<0.01	0.010	
Al-5Mg-1.0Cr		4.74		0.97		<0.01	0.010	
Al-5Mg-1.5Cr		4.92		1.39		<0.01	0.007	
Al-5Mg-1.5Mn		4.80			1.48	<0.01	0.011	
Al-5Mg-2.2Mn		4.80			2.24	<0.01	0.030	
Al-5Mg-0.3Y		6.26			0.25	<0.01	0.005	
Al-5Mg-0.6Y		5.30			0.62	<0.01	0.004	
Al-5Mg-1.2Y		5.16			1.22	<0.01	0.005	

(2) Observation of Recrystallization Behavior for As-extruded Materials

(TPR No.2; 1 Aug. 94 ~ 10 Oct. 94)

All powders were produced in an industry scale by Technical Research Laboratories of Sumitomo Light Metal Industries Ltd. in Japan. The atomized powders were sieved and the powders below 200 μm were used for extrusion.

Subsequently, the powders were compacted in an aluminum capsule with a diameter of 63.5 mm and degassed in a vacuum chamber (about 10^{-3} Torr) at 773 K for 3.6×10^3 ks. The compact billets were extruded into the bar of 6 mm thickness and 30 mm width with a reduction ratio of 26:1. The operating temperatures for Al-4.5wt% Cu and Al-5wt%Mg systems alloys were 653 and 703 K respectively.

The powder metallurgically processed aluminum alloys consist of two groups, statically recrystallized and dynamically recrystallized alloys. It is generally reported that dynamically recrystallized aluminum alloys exhibit superplasticity at higher strain rates than statically recrystallized aluminum alloys.

(3) Microstructural Evolution and Quantitative Metallographic Measurements

(TPR No.3; 10 Oct. 94 ~ 31 May. 95)

The detailed quantitative metallographic measurements of the size and the number of the second phase particles were carried out from the microstructures of as-extruded P/M Al-5wt%Mg systems alloys with various contents of Zr, Cr, Mn or Y. But, in cases for all materials, microstructural changes during straining at an optimum superplastic deformation condition could not be observed by the limited time, with the measurement of misorientation angles among grains and the dynamic behavior of the precipitates. I now strongly desire to continue this investigation of the microstructural changes in the near future by some funds from ARO.

(4) Experimental Measurements of Superplastic Properties and Grain Sizes

(TPR No.4; 1 June 95 ~ 31 July. 95: TPR No.5; 1 Aug. 95 ~ 30 Sep. 95:

TPR No.6; 1 Oct. 95 ~ 30 Nov. 95: TPR No.7; 1 Dec. 95 ~ 31 Jan. 96)

A determination of the flow characteristics and the elongation to failure in terms of the variation of strain rate with stress, temperature etc. was curried out

for all fourteen P/M aluminum alloys in a wide deformation conditions. Also the variation of grain size with testing temperature was investigated for all materials.

(5) Theoretical Interpretation to Superplastic Deformation Mechanisms

(TPR No.8; 1 Feb. 96 ~ 31 July. 96)

A theoretical interpretation, based on the experimental mechanical data by tensile test and the observed grain sizes, was considered in the form of the Dorn type constitute equation. Furthermore in order to test the new model of the accommodation process experimentally, the tensile tests and the EDS investigation and in-situ TEM observations at high temperatures near to the incipient melting point were performed for P/M aluminum alloys.

B. SUMMARY

This research leads to many important conclusions, are the following;

- All microstructures of as-extruded Al-Mg-Cr, Al-Mg-Mn and Al-Mg-Y systems alloys had already recrystallized statically, whereas those of Al-Cu-Zr and Al-Mg-Zr systems alloys with higher content of Zr remained unrecrystallized subgrain structures. The microstructures of Al-Cu-Zr and Al-Mg-Zr systems alloys with lower content of Zr consists of the mixing structures of large unrecrystallized subgrains and fine fully recrystallized grains.
- The sizes of the second phase particles (precipitates and inclusions) in Al-Mg alloys including Mn, Cr or Y are larger than those in another alloy with Zr. Also the size, the number and the volume of the second phase particles increase with content of Zr, Cr, Mn or Y. The distribution in the second phase particles for both Al-Mg and Al-Cu alloys with Zr is more inhomogeneous than that for the other alloys with Mn, Cr and Y.

- The detailed quantitative metallographic measurements from the microstructures of as-extruded P/M Al-5wt%Mg systems alloys with various contents of Zr, Cr, Mn or Y revealed that the size of the particles in all Al-Mg-Zr system alloys was one order magnitude finer than that in other Al-Mg system alloys with Cr, Mn or Y, and that the values in diameter for the maximum numbers of the particles were about 30 nm for the Al-Mg and Al-Cu system alloys with Zr, about 0.2 μm for the Al-Mg-Cr and Al-Mg-Y system alloys, and about 0.375 μm for the Al-Mg-Mn system alloys.
- The achieved minimum values in grain size with increasing of the contents of additional elements of Cr, Mn, Y and Zr to P/M aluminum alloys were about 1 μm for Al-Mg and Al-Cu systems alloys with Zr, about 3 μm for Al-Mg systems including Cr and Mn, and about 8 μm for Al-Mg-Y system alloys, as shown in **Fig. 1**. So an addition of Zr has a bigger contribution to improve a refinement of grain size in P/M Al-Mg system alloys than that of other elements of Mn, Cr and Y.

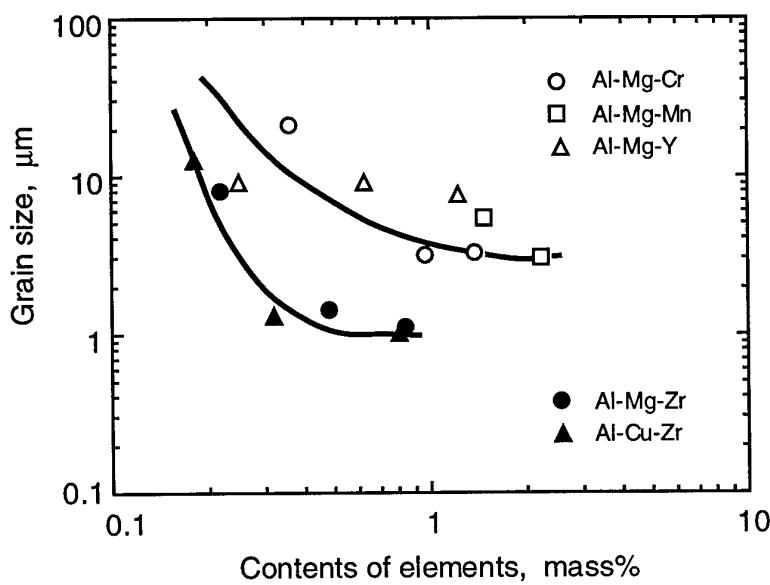


Fig. 1: The changes of the grain sizes in P/M aluminum alloys with the contents of additional elements of Cr, Mn, Y and Zr.

Table 2: Superplastic properties of P/M aluminum alloys.

Material	Temp. (K)	Strain Rate (s ⁻¹)	Stress (MPa)	m Value	Max. El(%)	Grain Size* (μm)
Al-4.5Cu-0.2Zr	798	10 ⁻⁴	4	0.4	100	15
Al-4.5Cu-0.3Zr	698	3x10 ⁻³	10	0.3	325	2
Al-4.5Cu-0.8Zr	773	10 ⁻²	8	0.3	600	1
Al-5Mg-0.2Zr	848	2x10 ⁻⁴	1	0.5	570	10
Al-5Mg-0.5Zr	748	10 ⁻¹	30	0.3	560	1
Al-5Mg-0.8Zr	773	10 ⁻¹	21	0.3	570	1
Al-5Mg-0.4Cr	848	10 ⁻⁴	2	0.5	240	30
Al-5Mg-1.0Cr	848	10 ⁻²	2	0.5	620	3
Al-5Mg-1.5Cr	848	10 ⁻²	3	0.5	510	3
Al-5Mg-1.5Mn	823	3x10 ⁻³	3	0.5	390	5
Al-5Mg-2.2Mn	823	6x10 ⁻³	4	0.5	570	3
Al-5Mg-0.3Y	848	10 ⁻⁴	0.5	0.5	570	10
Al-5Mg-0.6Y	823	4x10 ⁻⁴	2	0.5	435	9
Al-5Mg-1.2Y	848	10 ⁻³	1	0.5	775	8

* The values in grain size are obtained after annealing at superplastic temperatures for 30 min.

- Tensile tests revealed that all alloys exhibited superplasticity in a strain rate range from 10⁻⁴ to 10⁻¹ s⁻¹, as shown in **Table 2** and some materials, which had the grain sizes of less than 3 μm, exhibited high-strain-rate superplasticity between nearly 10⁻² and 10⁻¹ s⁻¹.
- Three Al-Mg-Cr, Al-Mg-Mn and Al-Mg-Y systems alloys, also Al-Cu-Zr and Al-Mg-Zr systems alloys with lower content of Zr with a statically recrystallized structure, were the statically recrystallized superplastic materials, whereas Al-Cu-Zr and Al-Mg-Zr systems alloys with higher content of Zr remained unrecrystallized subgrain structures were the dynamically recrystallized superplastic materials.
- An improvement in optimum superplastic strain rate for P/M aluminum alloys is resulting from the refinement of grains with increasing of Cr, Mn, Zr and Y contents. The result obtained in the present work seems to be consistent with

that reported in other aluminum alloys produced by the different processing routes, as shown in Fig. 2.

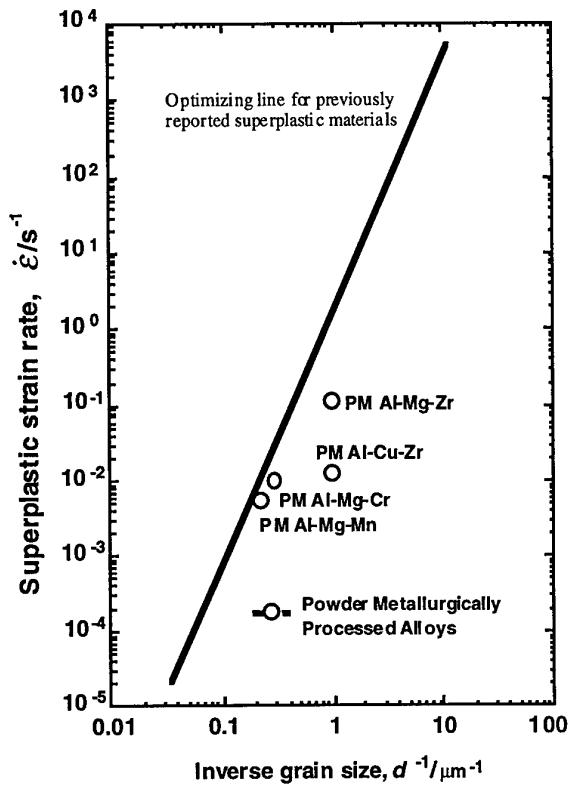


Fig. 2: Grain size dependence of superplastic strain rate in P/M aluminum alloys.

- The actively controlled processing methods in both size and volume among the second phase particles affected on the recrystallization behavior, the refinement of grain size and were very powerful to provide the desired structures required for high-strain-rate superplasticity in these P/M aluminum alloys.
- A theoretical interpretation revealed that values in activation energy for all alloys were between 140 and 155 kJmol⁻¹, which were similar to that for lattice self-diffusion of aluminum. Superplastic flow in these P/M aluminum alloys was fundamentally controlled by a grain boundary sliding mechanism accommodated by dislocation climb controlled by lattice self-diffusion.

- Each mechanical data of each statically or dynamically recrystallized superplastic alloy could be presented by a single equation, as shown in Fig. 3. The Dorn typed equation, given by the following;

$$\dot{\epsilon} = \frac{AGb}{kT} \left(\frac{b}{d} \right)^p \left(\frac{\sigma - \sigma_0}{G} \right)^n D_0 \exp\left(\frac{-Q}{RT}\right)$$

presented $n=2$, $p=2$ and $D=D_L$ (where $D=D_0 \exp(-Q/RT)$) for the statically recrystallized superplastic alloys consisted of the high-angle grain boundaries, whereas, $n=3$, $p=2$ and $D=D_L$ for the dynamically recrystallized superplastic alloys with the low-angle grain boundaries.

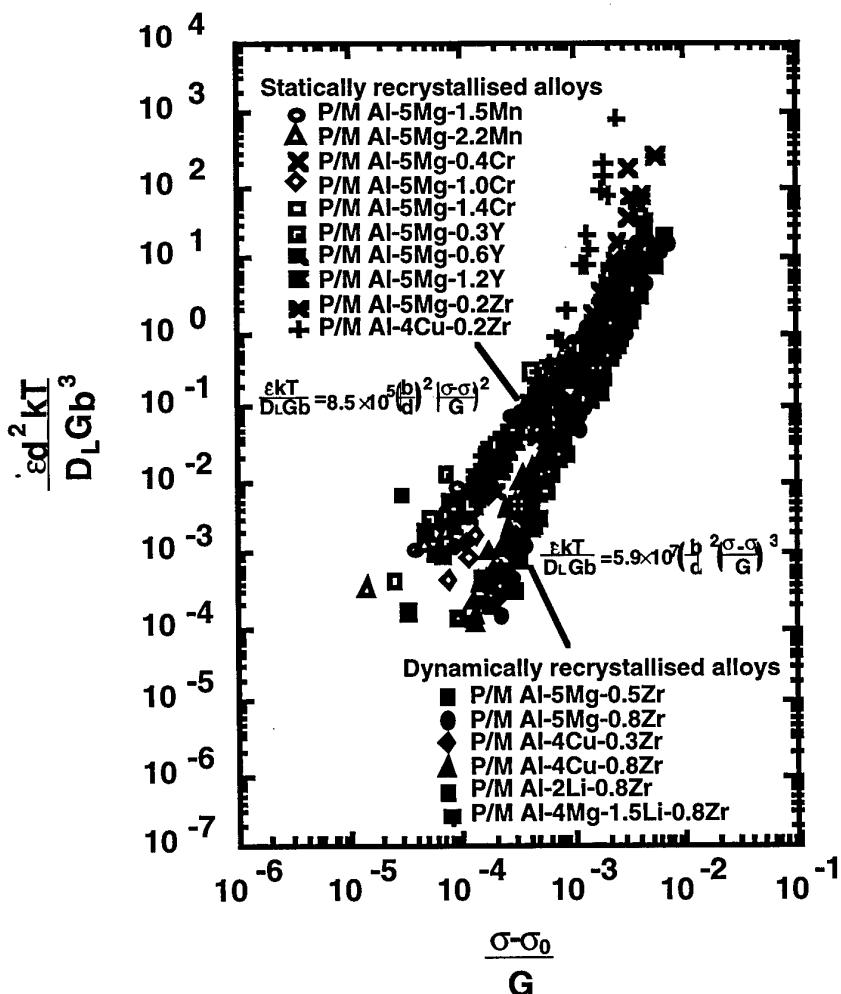


Fig. 3: Diffusion-compensated strain rate versus modulus-compensated stress for all P/M aluminum alloys.

- The consideration for deformation mechanisms with the accommodation helper by such a liquid has been applied on the data of the aluminum alloys, and the superplastic elongation strongly depended on the refinement of grain structures and the accommodation process by the presence of the helper, such as a liquid phase. But the contribution by an accommodation helper to high-strain-rate superplasticity remained unclear. A summary of the optimum superplastic temperatures, the solidus temperatures and the incipient melting temperatures of a number of P/M aluminum alloys is shown in **Table 3**.

Table 3; A summary of the optimum superplastic temperatures, the solidus temperatures and the incipient melting temperatures of a number of P/M aluminum alloys.

Material	Superplastic Temp.(K)	Solidus Temp.(K)	Incipient Melting Temp.(K)
Al-4.5Cu-0.2Zr	798	845	818
Al-4.5Cu-0.3Zr	698	847	818
Al-4.5Cu-0.8Zr	773	847	818
Al-5Mg-0.2Zr	848	852	
Al-5Mg-0.5Zr	748	852	
Al-5Mg-0.8Zr	773	852	
Al-5Mg-0.4Cr	848	854	
Al-5Mg-1.0Cr	848	856	
Al-5Mg-1.5Cr	848	855	
Al-5Mg-1.5Mn	823	847	
Al-5Mg-2.2Mn	823	843	
Al-5Mg-0.3Y	848	845	
Al-5Mg-0.6Y	823	843	
Al-5Mg-1.2Y	848	842	

- The simple and optimum thermo-mechanical treatment routes were established for P/M aluminum alloys, which will lead the cost effective production method for other materials could exhibit high-strain-rate superplasticity.
- A good understanding of the changes in mechanical properties during superplastic flow at high strain rates was provided, which could permit to develop the mechanism for high strain rate superplasticity based on microstructural view.

- It is still uncertain that the presence of the accommodation helper is unique requirement to achieve high-strain-rate superplasticity in P/M aluminum alloys, then it will be the further research subjects, including the investigation of cavitation and fracture.

C. LIST OF THE PUBLICATIONS

1. Effect of Temperature on the Mechanical Properties of Mechanically-Alloyed Materials at High Strain Rates
K.Higashi, T.G.Nieh and J.Wadsworth
Acta Metall. Mater., 43-9(1995) 3275-3282.
2. Mechanisms of High Strain-Rate Superplasticity of Al-14mass%Ni-14mass%Mm (Misch Metal) Alloy Produced from Amorphous Powders
K.Higashi, T.Mukai, A.Uoya, A.Inoue and T.Masumoto
Mater. Trans. JIM, 36, (1995-12) 1467-1475.
3. Interface Structure of Si₃N₄-Whisker-Reinforced Al-Mg-Si (Al alloy 6061) Composites Studied by High-resolution Electron Microscopy
H-G. Jeong, K. Hiraga, M. Mabuchi and K. Higashi
Philos. Mag. Lett. 74, No.2, (1996) 73-80.
4. A Quantitative Analysis of Cavitation in Al-Cu-Mg Metal Matrix Composites Exhibiting High Strain Rate Superplasticity
S.Wada, M.Mabuchi, K.Higashi and T.G.Langdon
J. Mater. Res., 11, (1996) 1755-1764.

5. Introduction to High Strain Rate Superplasticity

K.Higashi

Progress in Advanced Materials and Mechanics, ed. by W.Tzuchiang and T-W. Chou, Peking University Press, Beijing, (1996) 393-398.

6. High-strain-rate Superplasticity in Magnesium Matrix Composites Containing

Mg₂Si Particles

M. Mabuchi and K. Higashi
Philo. Mag. (1996) in press.

7. Strengthening Mechanisms of Mg-Si Alloys

M. Mabuchi and K. Higashi
Acta Mater. (1996) in press.

8. Superplasticity at a Low Temperature below 0.5Tm in an AZ91 Magnesium Alloy Processed by ECAE

Mabuchi, H. Iwasaki, K. Yanase, T. Mori and K. Higashi
Scripta Mater., (1996) in press.

9. High-Strain-Rate Superplasticity in Metallic Materials and Its Innovation in Ceramic Materials (Review)

Higashi, M. Mabuchi and T. G. Langdon
ISIJ International, 36, (1996) 1423-1438.

10. Partial Melting at Interfaces and Grain Boundaries for High Strain Rate Superplastic Materials

M. Mabuchi, H-G. Jeong, K. Hiraga and K. Higashi
Interfaces, (1996) in press.

11.Recent Works in High Strain Rate Superplasticity

K. Higashi and M.Mabuchi

Towards Innovation in Superplasticity, Mater. Sci. Forum, (1996) in press.

D. LIST OF PARTICIPATING SCIENTIFIC PERSONNEL

- Dr. Toshiji MUKAI

Researcher, Osaka Municipal Technical Research Institute, Joto-ku, Osaka 536,
Japan

- Mr. Mamoru MABUCHI

Researcher, National Industrial Research Institute of Nagoya, Hirate-cho,
Kita-ku, Nagoya 462, Japan

5. REPORT OF INVENTIONS: NONE

6. BIBLIOGRAPHY: NONE